OPTIMIZATION AND CHARACTERIZATION OF PECTIN EXTRACTED FROM SWEET POTATO RESIDUE

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ABSTRACT

Pectin has been used widely as thickener, stabilizer and gelling agent. However, the source of pectin industrially are still limited. This study aims to optimize the extraction of pectin from sweet potato residue using hydrochloric acid and analyses the properties of the pectin. In this study, the extraction of pectin from sweet potato residues using hydrochloric acid was optimized to maximise its yield and quality using response surface methodology (RSM). Then, the extracted pectin was further analyse for its degree of methylesterification (DM) using Fourier Transform Infrared Spectroscopy (FTIR). In this study, 0.5 %w/v, 1.0% w/v and 2.5% w/v concentration of pectin sugar solution were used to study their rheological properties. The antioxidant activity was assessed using DPPH radical scavenging activity. The optimum condition to extract pectin from sweet potato residues was at extraction temperature (60℃), pH (1) and extraction time (60 minutes) yielding 24.4 % pectin using hydrochloric acid. The pectin extracted has 57.48% of DM which indicated that it is high methoxyl pectin and able to form gel with the addition of sugar. The rheological properties analysis shows that 0.5% w/v pectin sugar solution shows Newtonian fluid behaviour while 1.0% w/v sugar pectin solution and 2.5% w/v pectin sugar solution showed pseudoplastic fluid behaviour. Moreover, the pectin was show 25.5 to 37.7% of radical scavenging activity.

ABSTRAK

Pektin telah digunakan secara meluas sebagai pemekat, penstabil dan ejen gelling. Walau bagaimanapun, sumber pektin dalam perindustrian masih terhad. Kajian ini bertujuan untuk mengoptimumkan pengeluaran pektin daripada sisa keledek menggunakan asid hidroklorik dan menganalisis sifat-sifat pektin. Dalam kajian ini, pengekstrakan pektin dari sisa keledek menggunakan asid hidroklorik telah dioptimumkan untuk memaksimumkan kaedah gerak balas permukaan hasil dan menggunakan kaedah (RSM). Kemudian, pektin yang diekstrak dianalisis dengan lebih lanjut untuk mengetahui darjah pengesteran (DM) menggunakan Fourier Transform Infrared Spektroskopi (FTIR). Dalam kajian ini, 0.5% w / v, 1.0% w / v dan 2.5% w / v kepekatan larutan gula pektin telah digunakan untuk mengkaji sifat-sifat reologi. Aktiviti antioksidan dinilai menggunakan aktiviti memerangkap radikal DPPH. Keadaan optimum untuk mengekstrak pektin dari sisa keledek adalah pada suhu pengekstrakan (60 °C), pH (1) dan masa pengekstrakan (60 minit) menghasilkan 24.4% pektin menggunakan asid hidroklorik. Pektin yang telah diekstrak mempunyai 57,48% daripada DM yang menunjukkan bahawa ia adalah pektin bermetoksil tinggi dan dapat membentuk gel dengan penambahan gula. Analisis sifat-sifat reologi menunjukkan bahawa 0.5% w / v larutan gula pektin menunjukkan kelakuan bendalir Newtonian manakala 1.0% w / v larutan gula pektin dan 2.5% w / v larutan gula pektin menunjukkan kelakuan bendalir pseudoplastic. Selain itu, pektin itu menunjukkan 25,5-37,7% daripada aktiviti memerangkap radikal.

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LIST OF ABBREVIATION

DM	-	Degree of Methyl Esterification
FT-IR	-	Fourier-transform Infrared Spectroscopy
HM	-	High Methoxyl
LM	-	Low Methoxyl
DPPH	-	1,1-diphenyl-2-picrylhydrazyl
RSM	-	Response Surface Methodology
CHE	-	Conventional Heating Extraction
MAE	-	Microwave assisted Heating Extraction
UAHE	-	Ultrasonic-assisted Heating Extraction

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Sweet potato, *Ipomoea batatas* L. (Lam.), a dicotyledonous plant that belongs to the family Convolvulaceae contains high dietary fibers, minerals, vitamin and antioxidant such as phenolic acid and carotene. It is also categorised as short term food crops. They are staple food crop in some developing countries. They are grown widely in tropical and subtropical region. Sweet potato also serves as animal feed and as raw product for industrial purposes. Moreover, sweet potato ranked as one of the most important crops in developing countries besides rice, wheat, maize and cassava (Hue & Low, 2015).

Pectin are natural hydrocolloids that are found in higher plants as main structural elements of cell walls. Basically, they are complex polysaccharides containing 1,4- linked-a-D galacturonic acid residues. Neutral sugar units are attached to the backbone and concentrated in highly branched ‘‘hairy” regions. Part of the carboxylic groups in the galacturonic chain exists in methyl ester form, and the degree of methylation (DM) divides pectin into two types. They are high methoxyl pectin (HM) and low methoxyl (LM) pectin. In the high-methoxyl (HM) form, more than 50% of the carboxyl groups are methylated, meanwhile in the low-methoxyl (LM) form less than 50% are methylated. The degree of methylation is very important for the gel formation of pectin.

Pectin is mainly used in food processing industry as gelling and thickening agents, emulsifiers as well as stabilizers in jams, jellies, confectionery products, and beverages. Other than that, pectin is also used in pharmaceutical and cosmetic industry as natural prophylactic, as binding agent in tablet formulations and as carrier of a variety of drugs. Pectin is also a component of soluble dietary fibres and plays a significant role in many physiological processes as it is poorly digested in the small intestine but ferments in the colon. For now, the source of commercial pectin are apple pomace and citrus peel, and to a certain extent sugar beet roots and sunflowers. However, there is a requirement for a novel source of pectin to manufacture pectin with tailored structures and beneficial properties, particularly adaptable for biomedical application. Therefore, the isolation of pectin from a cheap and abundant renewable resource, such as agricultural and food waste is a concern, moreover improve waste management and create another prospect of income to the economy.

The manufacture of pectin typically comprises extraction, purification, and drying. The use of a suitable method for pectin extraction is significant to maximize its yield and quality. Pectin can be extracted using various techniques. The most frequently used techniques are: i) solvent extraction by stirring and heating, ii) heat refluxing extraction and iii) microwave heating extraction. In solvent extraction of pectin by stirring and heating, different type of solvent was used. In this experiment, hydrochloric acid (HCl) was used as the solvent. The yield and properties of pectin usually depends on the raw materials used to extract pectin and the extraction conditions, such as temperature, extraction time, pH, and type of extraction solvents (Yeoh, Shi, & Langrish, 2008).

Traditionally, an optimization process has been conducted by monitoring the influencing of one factor at a time. However, the pectin extraction is a physical-chemical process in which hydrolysis and extraction of pectin macromolecules from plant tissue, and their solubilization take place continuously under the influence of different factors, mainly temperature, pH, and time. As such, the influence of these parameters, not only the single one but also the interaction between them, on the optimal extraction condition is necessary to be established. In order to provide a solution to this problem, the response surface methodology was consequently chosen as the tool to determine and to solve multivariable equations for optimizing the extraction processes of pectin from the sweet potato residues. The main objective of this study was to determine effects of the pectin extraction parameters (pH, temperature, and time) on their responses (yield). Such relationships finally led to an optimum condition for pectin extraction from sweet potato residue. In addition, the optimized sweet potato residue pectin was analysed for its degree of metyhlesterification, rheological properties and antioxidant properties.

1.2 Problem Statement

Sweet potatoes are mainly used to produce starch and starchy foods. Other than that, sweet potato also is being consumed directly. Thus, considerable quantity of residues is generated. Some of these residues are used as animal feed and the majorities of these residues are directly thrown out and consequently pollute the environment. The peels are mostly considered as waste materials. They are produced from the sweet potato processing industry and are normally discarded. Environmental problem, particularly these discarded peels of sweet potatoes can cause water pollution. Thus, in addition to being fed to animals, the peels can be utilized to produce pectin, which would then increase the potential return for the sweet potato processing industry.

Pectin is a complex polysaccharide that makes up about one third of the cell wall of higher plant. Pectin are found to be at its highest concentration in the middle lamella of cell wall. At present, commercial pectin are derived from citrus peel and apple pomace. They are by product from juice manufacturing. Apple pomace contains about 10- 15% of pectin on dry matter basis. Meanwhile, citrus peels contain relatively higher 20- 30% of pectin. Sugar beet waste from sugar manufacturing and sunflower head are alternatives sources of pectin. On the other hand, it is reported that sweet potato residues contain about 15 percent of pectin on its dry matter basis (Mei *et al*., 2010). Hence, sweet potato residue can be used as an alternative source of pectin.

The molecular size and degree of esterification (DE) of pectin play a vital role in the ability of pectin to from gel. Different sources of pectin do not necessarily have the same gelling ability due to variations in numerous parameters. Thus, the number of sources for commercial pectin manufacture is limited even though it occurs in most plant tissues (Srivastava & Malviya, 2011). In this study, the DE of pectin is identified using Fourier transform infrared (FTIR).

When many factors and interactions affect desired response, response surface methodology (RSM) is an effective tool for optimizing the process, which was originally described by Box and Wilson (1951). RSM is a collection of statistical and mathematical techniques that has been successfully used for developing, improving and optimizing processes. The main advantage of RSM is the reduced number of experimental trials needed to evaluate multiple parameters and their interactions. Therefore, it is less laborious and time-consuming than other approaches required to optimize a process (Giovanni, 1983). Response surface methodology has been successfully used to model and optimize the extraction of pectin from apple pomace (Wang *et al*., 2007), lemon byproduct (Masmoudi *et al*., 2008), passion fruit peel (Kliemann *et al*., 2009a), dragon fruit peel (Thirugnanasambandham *et al*., 2014), , grapefruit peel (Wang *et al*., 2015) and banana peels (Oliveira *et al*., 2016).

1.3 Objectives

This study embarks on the following objective:

1. To optimise the effect of acid, pH, temperature, and extraction time on yield of pectin from sweet potato residues (*Ipomoea batatas* var. Serdang 1) residues using response surface methodology.
2. To characterise the degree of methylesterification (DM), rheological properties and the antioxidant properties of pectin from sweet potato residues.

1.4 Scope of Study

1 Optimization of pectin yield extraction from sweet potato residue at different condition of pH (1-3), temperature (60- 90 ^{0}C) and time (30-90 minutes) using 0.1 M Hydrochloric acid (HCl) by Response Surface Methodology (RSM).

2 Determination of the degree of methyl esterification of extracted pectin using Fourier Transform Infrared (FTIR) Spectroscopy.

3 Analysing the rheological properties of the extracted pectin using viscometer at 0.5 %w/v, 1.0% w/v and 2.5% w/v concentration of pectin sugar solution.

4 Evaluation of antioxidant properties of pectin yield using 1,1-diphenyl-2-picrylhydrazyl (DPPH) analysis.

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